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IS 5295 (1985): Ethylene Glycol [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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Indian Standard
SPECIFICATION FOR
ETHYLENE GLYCOL
(*First Revision*)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR ETHYLENE GLYCOL

(First Revision)

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Indian Standard
SPECIFICATION FOR
ETHYLENE GLYCOL
(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 25 November 1985, after the draft finalized by the Organic Chemicals (Misc) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1969. The committee responsible for preparation of this standard decided to revise it in view of the growth in usage of the material in fibre and filament industry besides the general purpose use. The revised version covers two types of the material, namely, those catering to the need of the fibre and filament industry and those catering to the need of general purpose use such as antifreeze formulations, explosives and organic intermediates which are distinct from polyester fibres and filament industry. A separate specification for ethylene glycol antifreeze, namely IS : 5759-1970 has been prepared by Lubricants and Related Products Sectional Committee which covers requirements and the methods of sampling and test for antifreeze formulations.

0.3 Ethylene glycol is also known as 1, 2-ethanediol or more commonly as monoethylene glycol (MEG).

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Specification for ethylene glycol antifreeze.

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes requirements and the methods of sampling and test for ethylene glycol used in antifreeze formulations, explosives, organic intermediates and polyester fibres and filaments.

2. GRADE

2.1 There shall be two grades of the material, namely:

- a) General grade, and
- b) Fibre grade.

3. REQUIREMENTS

3.1 The material shall be colourless and free from suspended matter. It shall be miscible with water in all proportions at 20°C.

3.2 The material shall also comply with the requirements prescribed in Table I when tested according to the methods given in Appendix A. Reference to the relevant clauses of the appendix is given in col 5 of the table.

4. PRECAUTIONS IN HANDLING

4.1 The material shall be kept well protected from atmospheric humidity as it absorbs up to twice its mass of water vapour from the atmosphere.

5. PACKING AND MARKING

5.1 Packing — The material shall be packed in suitable and well closed containers with preferably, replaceable closure.

5.2 Marking — The containers shall be marked legibly and indelibly with the following information:

- a) Name and grade of the material;
- b) Net mass;
- c) Name of manufacturer and his recognized trade-mark, if any;
- d) Batch number; and
- e) Date of manufacture.

5.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

TABLE 1 REQUIREMENTS FOR ETHYLENE GLYCOL

(Clause 3.2)

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST, REF TO CL No. IN APPENDIX
		General Grade	Fibre Grade	
(1)	(2)	(3)	(4)	(5)
i)	Colour, Hasen units, <i>Max</i>	—	5	A-2
ii)	Colour after boiling for 4 hour under total reflux, Hazen Units, <i>Max</i>	—	10	
iii)	Relative density* at 27/27°C	—	1.110 2 to 1.110 7	A-3
iv)	Distillation range at 760 mm Hg:			A-4
	a) Initial boiling point, °C <i>Min</i>	195	195.5	
	b) The temperature at which 95 ml of material shall distil, °C	200	198.5	
v)	Moisture content, percent by mass, <i>Max</i>	0.3	0.2	A-5
vi)	Acidity (as acetic acid), percent by mass, <i>Max</i>	0.005	0.005	A-6
vii)	Ash, percent by mass, <i>Max</i>	0.005	0.005	A-7
viii)	Ultra-Violet transmittance in 1 cm cell at:			A-8
	a) 220 nm	—	60 percent	
	b) 275 nm	—	90 percent	
	c) 350 nm	—	98 percent	
ix)	Freezing point of equal volumes of material and water, <i>Max</i>	—30°C	—	A-9
x)	Iron content, ppm, <i>Max</i>	—	0.15	A-10
xi)	Chlorides content (as Cl), ppm, <i>Max</i>	—	0.3	A-11
xii)	Diethylene glycol content, percent by mass, <i>Max</i>	—	0.10	A-12

*Relative density is the term adopted for specific gravity with water as reference substance by the International Organization for Standardization (ISO).

6. SAMPLING

6.1 The procedure for sampling and the criteria for conformity of the material shall be as prescribed in Appendix B.

APPENDIX A

(Clause 3.2, and Table 1)

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. TEST FOR COLOUR

A-2.0 Outline of the Method — The colour of the material is compared with that of the colour standard and expressed in terms of Hazen colour units. The Hazen colour which is defined as the colour of an aqueous solution, containing 1 part per million of platinum in the form of chloroplatinic acid and 2 parts per million of cobaltous chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$).

A-2.1 Apparatus

A-2.1.1 *Nessler Cylinder* — Two, 100-ml capacity (see IS : 4161-1967†).

A-2.1.2 *One-Mark Volumetric Flasks* — 250-ml and 500-ml capacities (see IS : 915-1975‡).

A-2.2 Reagents

A-2.2.1 *Cobaltous Chloride Hexahydrate* — solid.

A-2.2.2 *Hydrochloric Acid* — Conforming to IS : 265-1976§.

A-2.2.3 *Chloroplatinic Acid* — Dissolve 250 mg of platinum in small quantity of aqua regia contained in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat this operation twice.

*Specification for water for general laboratory use (*second revision*).

†Specification for Nessler cylinders.

‡Specification for one-mark volumetric flasks (*first revision*).

§Specification for hydrochloric acid (*second revision*).

A-2.3 Preparation of Colour Standard — Dissolve 0.50 g of the cobaltous chloride hexahydrate and whole of the chloroplatinic acid in 50 ml of the hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into the 500-ml one mark volumetric flask. Dilute with water to the mark.

A-2.3.1 Pipette 2.5 ml/5.0 ml of the solution (*see* **A-2.3**) into two separate 250-ml one-mark graduated volumetric flask and dilute with water to the mark. The dilute solutions correspond to a colour of 5 and 10 Hazen units respectively and should always be freshly prepared.

A-2.4 Procedure — Fill one of the Nessler cylinders to the mark with the material to be tested, and the other with the colour standard. Compare the colours using a white background.

A-2.4.1 The material shall be taken to have passed the test if the colour of the test samples are not darker than that of the colour standard as required in Table 1.

A-3. DETERMINATION OF RELATIVE DENSITY

A-3.0 Outline of the Method — In this method, mass of equal volumes of the material and water are compared.

A-3.1 Apparatus

A-3.1.1 Relative Density Bottle — 25-ml capacity.

A-3.1.2 Water-Bath — Maintained at $27.0 \pm 0.2^{\circ}\text{C}$.

A-3.1.3 Thermometer — Any convenient thermometer of a suitable range with 0.1°C or 0.2°C sub-divisions.

A-3.2 Procedure — Clean and dry the relative density bottle weigh and then fill with recently boiled and cooled water at 27°C . Fill to overflowing by holding the relative density bottle on its stopper and immerse in the water-bath. Keep the entire bulb completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry and weigh. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

A-3.3 Calculation

$$\text{Relative density at } 27^{\circ}/27^{\circ}\text{C} = \frac{A - B}{C - B}$$

where

A = mass in g of the relative density bottle with the material;

B = mass in g of the relative density bottle; and

C = mass in g of the relative density bottle, with water.

A-4. DETERMINATION OF DISTILLATION RANGE

A-4.0 Outline of the Method — The material is distilled in the specified apparatus under the prescribed condition of heat input and rate of distillation. One hundred millilitres of the material are distilled and temperatures of initial boiling point and 95 percent volume recovery are recorded.

A-4.1 Apparatus

A-4.1.1 Distillation Flask — Shape and dimensions as shown in Fig. 1.

A-4.1.2 Thermometer — Partial immersion type, so fitted in the flask that the bottom of the capillary is level with the lower edge of the side-tube joint and the immersion mark is level with the top of the cork.

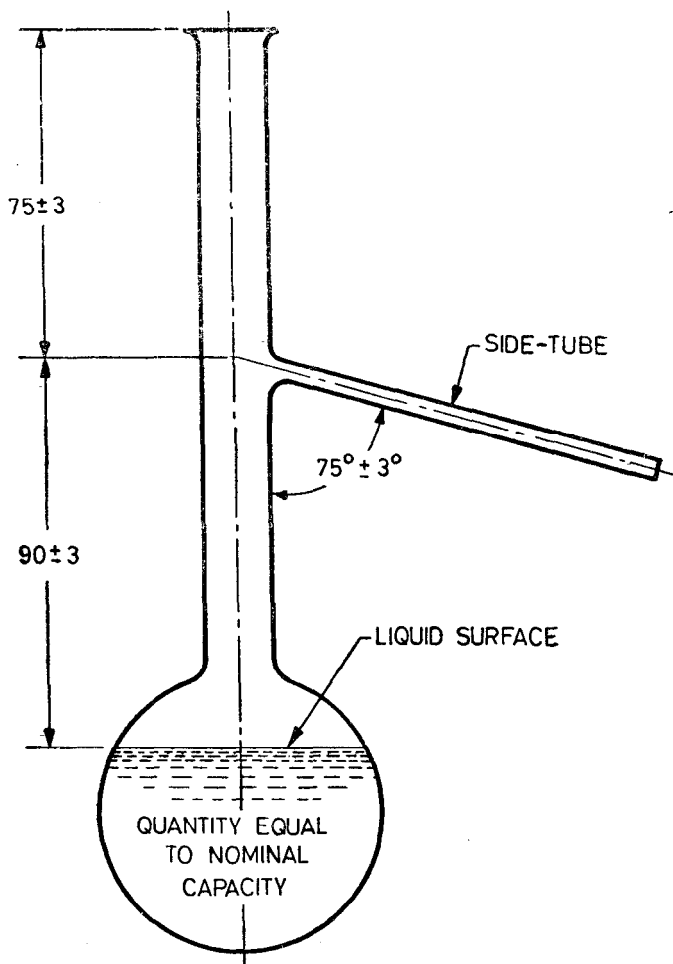
A-4.1.2.1 The recommended dimensions, tolerances and graduations of the thermometer are as follows:

Range	173 to 227°C
Graduation	0.2°C
Longer lines at each	1°C and 5°C
Fully figured at each	100°C
Fractional figuring at each	2°C and 10°C
Immersion	100 mm
Overall length	390 to 410 mm
Length of main scale	190 to 235 mm
Bulb length	15 to 20 mm
Stem diameter	6.0 to 8.0 mm
Distance from bottom of bulb to bottom of main scale	125 to 145 mm
Distance from bottom of bulb to top of contraction chamber, <i>Max</i>	35 mm
Maximum error	0.4°C

A-4.1.3 Rectangular Draught Screen — Rectangular in cross-section, made of 0.8 mm thick sheet metal, with dimensions as shown in Fig. 2 and open at the top and bottom. It shall comply with the following requirements:

- a) In each of the two narrower sides of the draught screen there shall be two circular holes each 25 mm diameter, and in each of the four sides of the draught screen there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the position shown in Fig. 2. The diameter of each of the holes centrally situated in the longer

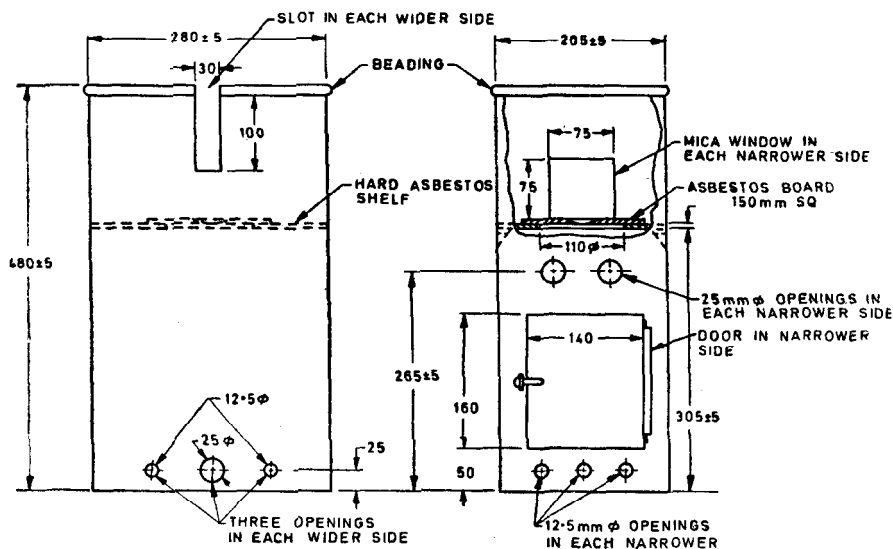
sides shall be 25 mm and of the remaining ten holes shall be 12.5 mm. At the middle of each of the wider sides a vertical slot with the dimension as shown in Fig. 2 shall be cut downwards from the top of the screen. A removable shutter conforming to the dimensions shown in Fig. 3 shall be provided for closing the vertical slot not in use.



All dimensions in millimetres.

FIG. 1 DISTILLATION FLASK

- b) A shelf of hard asbestos board, 6 mm in thickness and having a central circular hole 110 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.
- c) In one of the narrower sides of the screen a door shall be provided having dimensions and position as shown in Fig. 2. In each of the narrower sides of the screen a mica window shall be placed centrally with the bottom of the window on a level with the top of the asbestos shelf. The dimensions and position of the windows are shown in Fig. 2.
- d) An asbestos board 150 × 150 × 6 mm in size having a central hole 50 mm in diameter shall be so placed on the asbestos shelf that the two holes are approximately concentric and the distillation flask when in position completely closes the hole of the asbestos board.



All dimensions in millimetres.

FIG. 2 RECTANGULAR DRAUGHT SCREEN

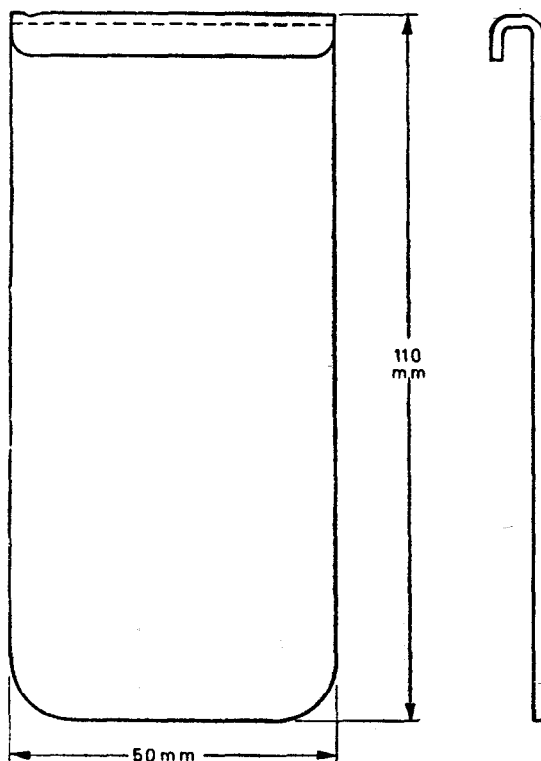


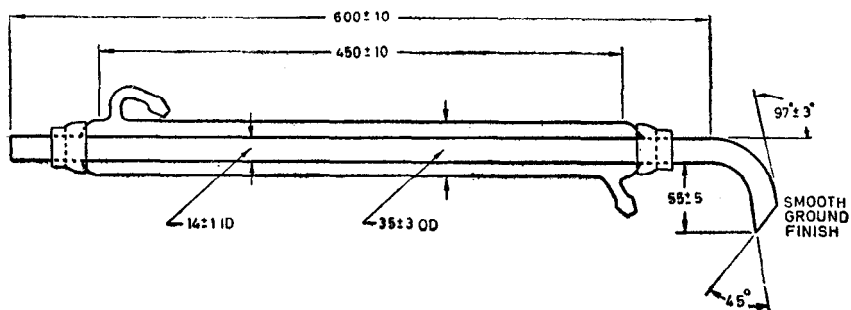
FIG. 3 REMOVABLE SHUTTER

A-4.1.4 Liebig Condenser — With the bent end made of Type 1 glass (graded according to IS : 2303-1963*), with a wall thickness of 1.0 to 1.5 mm and conforming to the shape and dimensions shown in Fig. 4. The bent portion may, however, be substituted by an adapter fitted externally in such a manner that distillate does not come in contact with the cork.

A-4.1.5 Receiver — of 100-ml capacity, with dimensions and graduation as shown in Fig. 5.

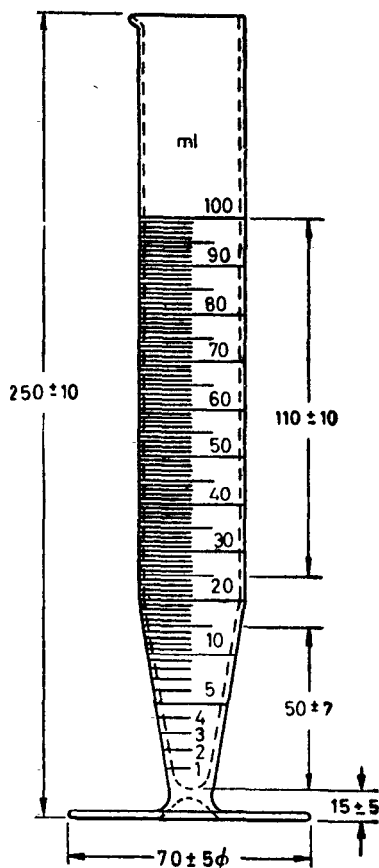
A-4.1.6 Electric Heater, Gas Burner or Other Flame Type Heater — Any suitable heater or burner that enables the distillation to be carried out as described in A-4.2.

*Method of grading glass for alkalinity.



All dimensions in millimetres.

FIG. 4 LIEBIG CONDENSER



All dimensions in millimetres.

FIG. 5 RECEIVER

A-4.2 Procedure — Assemble the apparatus as shown in Fig. 6. Measure 100 ml of the material at laboratory temperature by means of the receiver and transfer it to the distillation flask. Add a fragment (about 2 mm cube) of porous or other suitable inert material to prevent bumping, connect the flask to the condenser and insert the thermometer. Fit the thermometer in the flask so that the bottom of the capillary is in level with the lower edge of the side-tube joint. Pass an adequate supply of water.

To receive the distillate, use the receiver in which the sample was measured, without rinsing or drying. Heat the flask slowly; especially after ebullition has begun, in order that the mercury column of the thermometer may become fully expanded before the first drop of distillate passes into the receiver, care being taken that the total period of this preliminary heating shall be not less than 5 nor greater than 10 minutes. Place the receiver in a manner that the condensate flows down its side. Continue the distillation at the rate of 4 to 5 ml per minute (about 2 drops per second). Read on the thermometer when initial boiling point is reached and 95 ml of distillate have been collected in the cylinder.

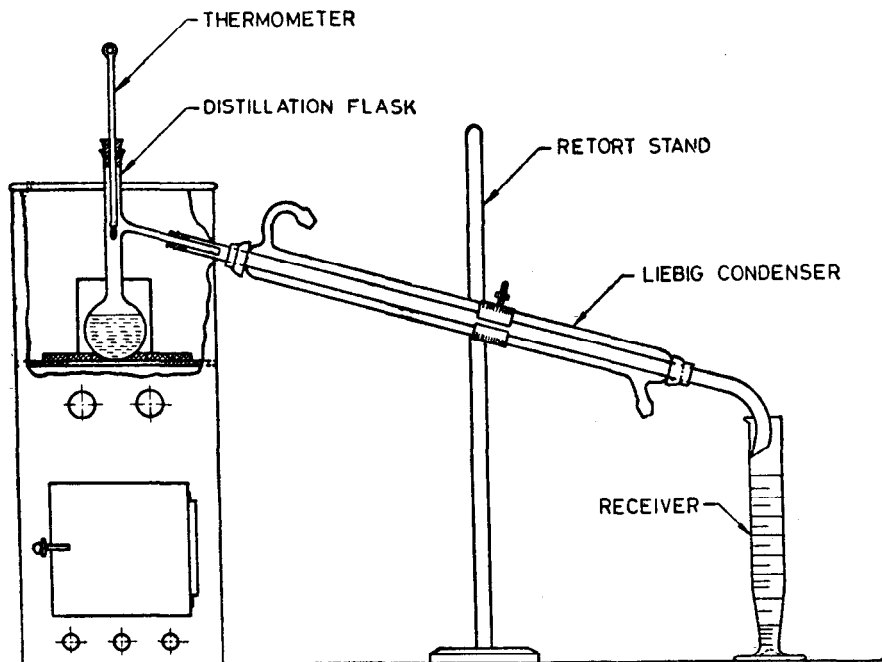


FIG. 6 ASSEMBLY OF APPARATUS FOR DETERMINING DISTILLATION RANGE

A-4.3 Correction of the Thermometer Reading

A-4.3.1 Error of Scale — In all the thermometer readings, make the corrections as indicated on the certificate of the instrument.

A-4.3.2 Correction for Barometric Pressure — If the barometric pressure prevailing during the determination is normal, namely, 760 mmHg, no correction need be applied to the specified temperature and the thermometer scale as corrected under **A-4.3.1** shall be used as such. If, however, the prevailing pressure deviates from 760 mmHg, the specified temperatures shall also be corrected as follows:

- a) For every 10 mmHg above 760 mmHg subtract 0.43°C from the specified temperature; and
- b) For every 10 mmHg below 760 mmHg, add 0.43°C to the specified temperature.

NOTE — These corrections are valid only for pressures above 700 mmHg.

A-5. DETERMINATION OF MOISTURE CONTENT

A-5.0 General — Moisture is determined by the Karl Fischer method.

A-5.1 Procedure — Take about 10 g of the material, weigh accurately and determine the moisture content by the procedure given in IS : 2362-1973*.

A-6. DETERMINATION OF ACIDITY (AS ACETIC ACID)

A-6.0 Outline of the Method — The material is titrated with standard sodium hydroxide solution using phenolphthalein as indicator.

A-6.1 Reagents

A-6.1.1 Phenolphthalein Indicator — 0.5 percent (*m/o*). Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit (*see* IS : 323-1959†) and make it faintly pink by the addition of dilute sodium hydroxide solution.

A-6.1.2 Standard Sodium Hydroxide Solution — 0.01 N.

A-6.2 Procedure — Weigh accurately about 100 g of the material into a 500-ml conical flask to the nearest 0.1 g. Pour about 100 ml of freshly boiled and cooled distilled water and add 2 to 3 drops of phenolphthalein indicator. Mix well and note the colour of the solution. A pink colouration indicates the presence of alkalinity. If the solution is colourless, titrate with standard sodium hydroxide solution until a pale pink colour persists for more than 15 seconds.

*Determination of moisture by Karl Fischer method (*first revision*).

†Specification for rectified spirit (*first revision*).

Take 100 ml of distilled water in another 500 ml conical flask and carry out blank test.

A-6.3 Calculation

$$\text{Acidity (as acetic acid),} \\ \text{percent by mass} = \frac{(V_1 - V_2) \times N \times 0.06}{M} \times 100$$

where

V_1 = volume in ml of standard sodium hydroxide solution used in the test with the material,

V_2 = volume in ml of standard sodium hydroxide solution used in blank test,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

A-7. DETERMINATION OF ASH

A-7.0 General — The material is heated at 600°C to 700°C and ash determined.

A-7.1 Procedure — Weigh accurately about 100 g of the material into a tared platinum or silica dish. Heat gently at first, evaporate to dryness and then ignite to almost ash. Then heat in an electric furnace at 600°C to 700°C for about 30 min, allow to cool in a desiccator and weigh. Repeat the heating and cooling till the difference between two successive weighings falls within 0.5 mg.

A-7.2 Calculation

$$\text{Ash, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the ignited residue, and

M_2 = mass in g of the material taken for the test.

A-8. DETERMINATION OF UV TRANSMITTANCE

A-8.0 General — The UV transmittance of ethylene glycol is a measure of purity of the product. The transmittance of a sample is measured at 220, 275 and 350 nm on the Perkin Elmer Lambda 3 Spectrophotometer. Low transmission at any wave length indicates low purity with the wave length being characteristic of the impurity.

A-8.1 Apparatus

A-8.1.1 *Perkin Elmer Lambda 3 Spectrophotometer*

A-8.1.2 Ultra-violet Light Source

A-8.1.3 Silica Cells — one centimetre

A-8.2 Reagents

A-8.2.1 Distilled Water — See A-1.1.

A-8.3 Procedure — Inspect all equipment to ensure operability and the cells to insure that they are absolutely clean and dry. Prepare the spectrophotometer for use with the ultraviolet light source. Adjust transmission to 100 percent with distilled water in cell (against distilled water reference for double beam operation) at 220 nm. Fill the silica cell (insuring cell is dry before use) with ethylene glycol and the reference cell with distilled water. Place the cells in the cell holder and the cell holder in spectrophotometer. Determine the transmittance of the sample at 220 nm, 275 nm and 350 nm.

A-8.4 Report — Report results as percent transmission of virgin ethylene glycol at 220 nm, 275 nm and 350 nm.

A-9. DETERMINATION OF FREEZING POINT

A-9.1 Preparation of Test Sample — Mix thoroughly equal volumes of the material and water.

A-9.2 Procedure — Determine the freezing point of the test sample following Method B of IS : 1448 (P : 11)-1971* except that the sample shall not be dried before determining the freezing.

A-10. DETERMINATION OF IRON CONTENT

A-10.0 General — In alkaline medium, thioglycolic acid gives a reddish violet solution with ferrous and ferric irons. In this method the material is treated with thioglycolic acid and ammonia and the colour produced compared with that produced by standard iron solution under identical conditions.

A-10.1 Apparatus

A-10.1.1 Nessler Cylinders — 100-ml capacity (see IS : 4161-1967†).

A-10.2 Reagents

A-10.2.1 Thioglycolic Acid Solution — Dissolve 10 g of thioglycolic acid in distilled water to make 100 ml solution.

A-10.2.2 Concentrated Ammonia — relative density 0.90.

*Methods of test for petroleum and its products: P : 11 Freezing point (second revision).

†Specification for Nessler cylinders.

A-10.2.3 Standard Iron Solution — Weigh 0.702 g of ferrous ammonium sulphate and dissolve in 100 ml of water. Make up 1 ml of this solution to 100 ml with water. One millilitre of this solution then contains 0.01 mg of iron.

A-10.3 Procedure — Weigh accurately 50 g of the material into a Nessler cylinder, pour 25 ml of water, add 2.5 ml of thioglycolic acid solution and 5 ml of concentrated ammonia. Mix thoroughly and make up the volume to 100 ml with water. Take, in another Nessler cylinder, 75-ml, distilled water and 1 ml of standard iron solution, add 2.5 ml of thioglycolic acid solution and 5 ml of concentrated ammonia. Mix thoroughly, make up the volume to 100 ml with water and after 10 minutes compare the colours of both solutions.

A-10.3.1 The iron content of the material shall be regarded as not having exceeded the limit prescribed in Table 1 if the intensity of colour produced with the material is not deeper than that produced with standard iron solution.

A-11. DETERMINATION OF CHLORIDES (As Cl)

A-11.0 Outline of the Method — Turbidity of the solution is compared with the same quantity of standard chloride solution in Nessler cylinders.

A-11.1 Apparatus

A-11.1.1 Nessler Cylinders — 50-ml capacity.

A-11.2 Reagents

A-11.2.1 Concentrated Nitric acid — See IS : 264-1976*.

A-11.2.2 Silver Nitrate Solution — Approximately 2 percent (*m/v*).

A-11.2.3 Standard Chloride Solution — Dissolve 1.649 g of sodium chloride in water and make up the volume to exactly 1 000 ml. Pipette out 10 ml of the solution, dilute with water and again make up the solution to exactly 100 ml. One millilitre of this solution contains 0.1 mg of chloride (as Cl).

A-11.2.4 Phenolphthalein Indicator Solution — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-11.3 Procedure — Weigh 10.0 g of the material, dissolve in 15 ml of water, add a drop of phenolphthalein indicator and neutralize cautiously with concentrated nitric acid. If the solution is not clear, filter through a filter paper Whatman No. 40 or 42 that has been washed free of chloride and dilute the filtrate to exactly 50 ml in a volumetric flask.

*Specification for nitric acid (*second revision*).

A-11.3.1 Take 25 ml of aliquot in a Nessler cylinder add 1 ml of silver nitrate solution and dilute to 50-ml mark. Carry out a control test in the other Nessler cylinder using 1 ml of the standard chloride solution and the same quantities of other reagents and finally diluting to 50-ml mark. Stir both the solutions with glass rod and compare the turbidity produced in the two cylinders after 5 minutes.

A-11.3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with material is not greater than that produced in the control test.

A-12. DETERMINATION OF DIETHYLENE GLYCOL IN ETHYLENE GLYCOL

A-12.0 General — The chromatographic conditions given here are for guidance only. Variations in conditions to arrive at the typical chromatographic separation shown in Fig. 7 are possible.

A-12.1 Outline of the Method — A sample of the material is injected into the gas chromatograph from where it is carried by the carrier gas from one end of the column to the other end. During its movement the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after the other and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

A-12.2 Apparatus

A-12.2.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. A typical chromatogram using such a chromatograph is shown in Fig. 7 which may be obtained with the following chromatographic conditions:

<i>Column</i>	Material: glass or stainless steel Length \times ID : 1 100 \times 2 mm Stationery phase: 10 percent W Carbowax 20 M Solid support: Chromosorb G-AW-DMCS, 60/80 mesh Ratio : Stationery phase/solid support (1/9)
<i>Carrier Gas</i>	Hydrogen
<i>Conditions</i>	Column temperature — Isothermal 165°C Injection port temperature 200°C Carrier gas flow rate — 2.4 l/h (Nitrogen)
<i>Detector</i>	Type: Flame Ionisation, Temp: 200°C
<i>Sample</i>	10 μ l (1 : 1 dilution by volume with acetone on column injection)

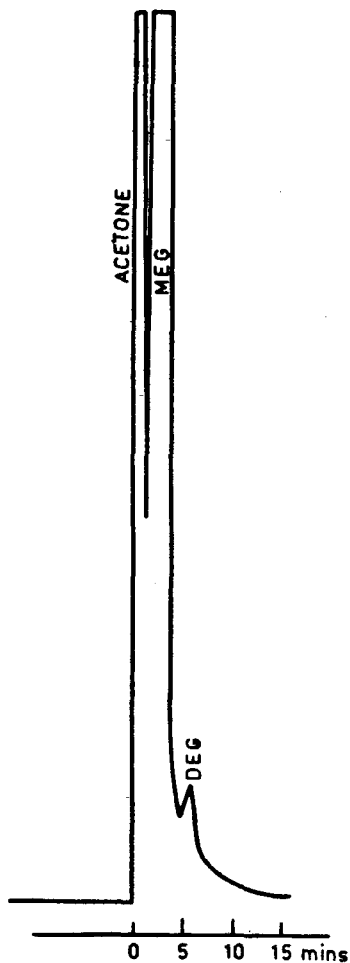
A-12.3 Procedure — Conduct the flow of the carrier gas and inject sample at injection port where it is vaporized and well mixed with carrier gas. This is led into the chromatographic column wherein vaporized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. For this separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents. As the sample enters the detector, it gives the signal corresponding to the amount of particular constituents leaving the column. The detector signals, on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

A-12.4 Calculation

A-12.4.1 Calculate, the peak areas of individual constituent pertaining to diethylene glycol on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure diethylene glycol using the same apparatus under identical conditions.

A-12.5 Results

A-12.5.1 The typical chromatogram is shown in Fig. 7.



Column : 1 100 mm \times 2 mm SS tube with 10 percent W Carbowax 20 M on Chromosorb G-AW-DMCS, 60/80 mesh

Oven : 165°C
Injection port : 200°C
Detector : 200°C
Carrier gas : N₂, 2.4 l/hr
Sample size : 10/ μ l (1 : 1 ml with acetone)

FIG. 7 TYPICAL CHROMATOGRAM FOR DETECTION OF DEG IN MEG

A P P E N D I X B

(Clause 6.1)

SAMPLING OF ETHYLENE GLYCOL

B-1. GENERAL REQUIREMENTS

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry and shall be made of low or reduced spark generating material.

B-1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination (*see also 4.1*).

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

B-1.5 The samples shall be placed in suitable, clean, dry and airtight glass containers preferably of amber or blue colour.

B-1.6 The sample containers shall be of such a size that they are almost but not completely, filled by the sample.

B-1.7 Rubber stoppers or composition corks shall not be used for closing the sample bottle.

B-1.8 Sealing wax or other plastic material, if used, shall be applied in such a way that it does not contaminate the sample when the bottles are opened.

B-1.9 Each sample container shall be protected by covers of oil-proof paper, metal foil, viscose or other suitable impervious material over the stopper to keep away moisture and dust from the mouth of the bottle and to protect it while being handled.

B-1.10 Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling, such as the date of sampling, the year of manufacture of the material, the batch number, the name of the supplier, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample.

B-2. SAMPLING INSTRUMENT

A-2.0 The following forms of sampling instruments may be used:

- a) Sampling bottle or can, for taking samples from various depths of large tanks; and
- b) Sampling tube.

B-2.1 Sampling Bottle or Can — It consists of weighed bottle or metal container with removable stopper or top, to which is attached a light chain (*see* Fig. 8). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

B-2.2 Sampling Tube — It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 9). The ends are conical and reach 5 to 10 mm diameter at the tips. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

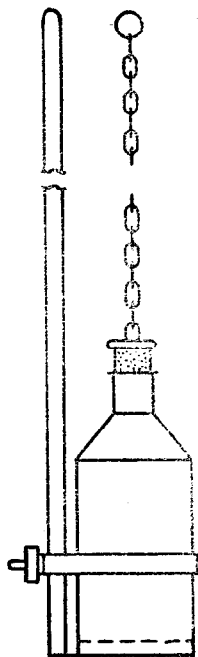
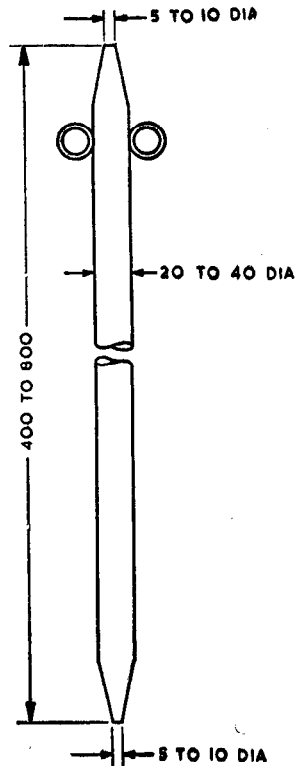


FIG. 8 SAMPLING BOTTLE OR CAN



All dimensions in millimetres.

FIG. 9 SAMPLING TUBE

B-2.2.1 For small containers, the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 Lot — In any consignment, all the containers of the same size and drawn from same batch of manufacture shall constitute a lot.

B-3.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 SCALE OF SAMPLING

(Clause B-3.2)

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
(<i>N</i>)	(<i>n</i>)
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 300	6
301 to 500	7
501 and over	8

NOTE 1 — In case of large-sized containers, such as tanks, tank lorries or tankers, every container in the lot shall be sampled separately.

NOTE 2 — In case of very small lots where the selection of three containers may be uneconomical, the number of containers to be selected and the method of judging the conformity of the lot to the requirements of this specification shall be as agreed to between the purchaser and the supplier.

B-3.3 The containers shall be chosen at random from the lot with the help of a suitable random number table. In case no such table is available the following procedure is recommended for use:

Starting from any container of the lot, count them as 1, 2, 4... up to r and so on in one order, where r is the integral part of N/n (N being the number of containers in the lot and n the number of containers to be selected). Every r th container thus counted shall be withdrawn to give sample for tests.

B-4. PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected according to **B-3.2** and **B-3.3**, small portions of the material shall be drawn with the help of the suitable sampling instrument (see **B-2**) from several positions at the upper, middle and bottom layers according to Table 3. These portions shall be mixed together so as to yield a quantity of material not less than 1 000 ml representative of that container.

From every large-sized container, a quantity of material not less than 1 000 ml representative of the container shall be made up of portions drawn according to Table 3.

TABLE 3 DEPTHS OF SAMPLING LAYER AND PROPORTIONS OF THE SAMPLE

(Clause B-4.1)

DEPTH OF CON- TENT (PERCENT) OF THE DIAM- ETER	DEPTHS OF SAMPLING LAYER (HEIGHT FROM BOTTOM, PER- CENT OF THE DIAMETER			PROPORTIONS OF THE SAMPLE TO BE DRAWN		
	Upper Layer	Middle Layer	Bottom Layer	Upper Layer	Middle Layer	Bottom Layer
(1)	(2)	(3)	(4)	(5)	(6)	(7)
100	80	50	20	3	4	3
90	75	50	20	3	4	3
80	70	50	20	2	5	3
70	—	50	20	—	6	4
60	—	50	20	—	5	5
50	—	40	20	—	4	6
40	—	—	20	—	—	10
30	—	—	15	—	—	10
20	—	—	10	—	—	10
10	—	—	5	—	—	10

B-4.2 From each of these individual samples, an equal quantity of material shall be taken and mixed thoroughly to form a composite sample, of about 1 000 ml. The composite test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4.3 The parts of the composite test sample, obtained according to **B-4.2**, shall be transferred to separate containers and shall be sealed and marked with full identification particulars given in **B-1.10**.

B-4.4 The reference test sample shall bear the seals of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier to be used in the case of any dispute between the two.

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to the requirements of this specification if the composite sample does not fail to satisfy any of the specified requirements.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²



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